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TECHNICAL REPORT 4152

FAILURE OF ADHESIVE BONDS AT CONSTANT STRAIN RATES

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MARCH 1971

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PICATINNY ARSENAL
DOVER, NEW JERSEY

Technical Report 4152

FAILURE OF ADHESIVE BONDS AT CONSTANT STRAIN RATES

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OBJECT

The object of this work was to study the shear strength of a structural ahesive used in Army helicopters as a function of strain rate and failure time at several temperatures.

SUMMARY

Shear strengths of adhesive AF126 bonds to two thicknesses of aluminum were measured at constant rate of crosshead separation. Primarily cohesive failure was observed in all cases. By using relations proposed by Cherry and Holmes, the shear strength could be quantitatively related to temperature and strain rate or failure time. Of specific engineering interest would be the possibility of determining the reliability of a bonded joint for specific periods of time as a function of continuously applied stress at a given temperature.

INTRODUCTION

Scientists in the Materials Engineering Laboratory at Picatinny Arsenal have in the past shown some interest in the causes of failure of adhesive bonds under various loading conditions (Refs 1, 2). More recently, some constant strain rate shear strength determinations have been made on an AF126 adhesive used for structural bonding in Army helicopters. This paper shows that the shear strength of such a bond, under conditions where cohesive failure occurs, can be quantitatively related to temperature and strain rate or failure time.

RESULTS AND DISCUSSION

Tables 1 and 2 show the data for the constant strain rate (constant crosshead separation) measurements on 1/8 inch and 1/16 inch aluminum adherends bonded with AF126 adhesive. Three crosshead rates (0.105, 0.8, and 2 in. per min) were used at each of six temperatures (193^o, 233^o, 273^o, 296^o, 323^o, and 343^oK).

Cherry and Holmes (Ref 3) suggested that when a shear stress is applied to a polymer there is an instantaneous elastic deformation resulting from bond angle bending as well as simultaneous plastic deformation, stretching the polymer chains joining adjacent slipped

areas of the polymer until these chains break. When a critical rate of chain scission is exceeded, catastrophic failure occurs. For constant strain rate experiments, they obtained the relation

$$S = (2kT/\beta)\ln C + (2kT/\beta)\ln(hl/\rho\beta RT) + (2RG_e)^{1/2} + 2\varepsilon_o/\beta \quad (1)$$

S is stress at failure, β is volume of elements that respond in failure, C is rate of crosshead separation, l is diameter of "dislocation loop", ρ is density of dislocation lines in unit volume of the medium, $(2RG_e)^{1/2}$ is a critical value of strain at failure, and ε_o is an activation energy barrier. Since the last three terms on the right-hand side of Equation 1 are constant at a given temperature, this equation requires that a plot of S versus log C be linear. Figures 1 and 2 show such plots for the data given in Tables 1 and 2.

An alternative form of Equation 1 may be obtained by dividing through by T

$$S/T = (2k/\beta)\ln C + \frac{2k}{\beta}\ln\left(\frac{hl}{\rho\beta RT}\right) + \frac{(2RG_e)^{1/2}}{T} + \frac{2\varepsilon_o}{\beta T} \quad (2)$$

In this case, the S/T vs log C plots should be linear for each temperature. Figures 3 and 4 show the appropriate straight lines.

Equations 1 and 2 suggest that S or S/T should also be linear with $\log(1/t_f)$. This will be so if the deformation (ε) is constant at a given temperature. (t_f is failure time.)

$$Ct_f = \varepsilon \quad (3)$$

The Ct_f products shown in Table 3 give an indication of the constancy of ε at each temperature. For 1/16 inch aluminum in the three cases where values are not recorded, the machine stalled on release, leading to a hesitation and longer times than would otherwise have been observed. A somewhat more convenient representation can be given by rearranging Equation 3 and taking logarithms.

$$\log C = \log\varepsilon + \log(1/t_f) \quad (4)$$

Equation 4 shows that a plot of log C versus log $(1/t_f)$ drawn with a slope = 1 will yield ε at log $(1/t_f) = 0$. Figures 5 and 6 show the straight lines drawn with slope = 1 in each case. Values of log ε obtained from these lines are given in Table 4.

It is interesting to note that there appears to be a small but noticeable variation of deformation with temperature for the samples using 1/8 inch aluminum adherends. In the case of 1/16 inch aluminum adherends, the deformation does not appear to vary with temperature, at least beyond the range of experimental error. The deformation (as used in this report and calculated from the observed C and t_f values) is presumably made up of grip slippage, and deformation in the links of the machine, the aluminum metal adherends, and the adhesive. The 1/16 inch aluminum adherends deform visibly under the test conditions while the 1/8 inch aluminum adherends do not. The observed differences are probably due to this difference in the response of the aluminum.

The reasonable constancy of the deformation at a given temperature for each of the crosshead rates makes it possible to replace log C in either Equation 1 or Equation 2 with log ε + log $(1/t_f)$. Then at a given temperature S(or S/T) should be linear with log $(1/t_f)$.

$$S = \frac{(2kT)}{\beta} \ln\varepsilon + \frac{(2kT)}{\beta} \ln\frac{(1)}{t_f} + \frac{2kT}{\beta} \ln\frac{(h1)}{\rho\beta kT} + (2RG_e)^{1/2} + \frac{2\varepsilon_o}{\beta} \qquad (5)$$

or

$$\frac{S}{T} = \frac{(2k)}{\beta} \ln\varepsilon + \frac{(2k)}{\beta} \ln\frac{(1)}{t_f} + \frac{2k}{\beta} \ln\frac{(h1)}{\rho\beta kT} + \frac{(2RG)^{1/2}}{T} + \frac{2\varepsilon_o}{\beta T} \qquad (6)$$

Figures 7-10 show that the lines required by Equations 5 and 6 are reasonably linear.

Equations 1, 2, 5, and 6 clearly indicate that the slopes of the lines in Figures 1-4, and 7-10 permit evaluation of β. Table 5 gives values of β determined from each of the four plots at each thickness of aluminum adherend. Average values are summarized in Table 6. Although numerical values of β for the crosslinked modified epoxy are considerably lower than those found by Cherry and Holmes (Ref 3) for polyethylene, β increases with temperature as observed by those investigators.

Some constant stress data for AF126 adhesive used with 1/16 inch aluminum panels is available (Ref 2). In this case, Cherry and Holmes (Ref 3) obtained the relation

$$S = \frac{(-2kT)}{\beta} \ln t_f + \frac{(2kT)}{\beta} \ln \frac{(h1\gamma_f)}{\rho\beta kT} + (2RG_e)^{1/2} + 2\varepsilon_o/\beta \qquad (7)$$

Equation 7 requires that S be linear with log t_f with β readily accessible from the slope as in the case of constant rate of strain experiments. Such a comparison should be useful in giving an indication that the same parameters describe the behavior under differing types of loading. Unfortunately, a direct comparison is not possible since at the long times of the constant stress experiments, the failure time was rather markedly dependent on humidity (Ref 2). Also, at the much lower loads of the constant stress experiments, there was no noticeable deformation of the 1/16 inch aluminum. Hence, a quantitative comparison of β values for the two cases does not seem to be in order. However, Figures 11-13 show that the constant stress data, allowing for the expected adhesive scatter, does obey Equation 7. This gives some confidence that these relations can describe data obtained by different loading methods. Table 7 gives apparent β values calculated from the slopes of the lines in Figures 11-13. Qualitatively, the values appear to be in the general range that would be expected from the results described above. However, in each case, there seems to be a rather sharp and unexpected downturn in the β (increase in slope) curve at the highest temperature (344°K).

EXPERIMENTAL PROCEDURE

Materials

2024-T3 aluminum panels, 4" x 13" x 1/8".

2024-T3 aluminum panels, 4" x 12" x 1/16".

AF126-3, a thermosetting, nonvolatile, modified epoxy film adhesive designed for structural bonding of metals.

Preparation of Adherends

Scribe marks were placed 1/2 inch from the long edge of each panel to assure an accurate overlap of the joint. Two panels were then clamped together in the position for bonding using the scribe marks as a guide. Alignment holes were drilled through the area to be bonded at both ends of the panel. The clamps were then removed and the panels were washed with acetone followed by degreasing in hot vapors of stabilized perchloroethylene. The area to be bonded was then etched for 5 minutes at 150°F in FPL etch solution in accordance with MIL-A-9067C, washed with tap water at 140°F, and rinsed in deionized water. The panels were dried in a forced-air circulating oven at 140°F for one hour.

Preparation of Lap Shear Specimens

One group of panels was prepared using the 1/16 inch thick aluminum adherends and a second group was prepared using the 1/8 inch thick adherends.

A single layer of the film adhesive was placed in the joint and the two adherend panels were pinned in place by putting aluminum rods through the previously drilled holes and hammering the protruding ends flat. Three bonded panels were prepared at one time, overlapping the ends to give them proper support. An extra panel was used for the same purpose under the end of the third bonded panel. The assembly was placed in a hydraulic press at room temperature and subjected to 50 psi pressure. The temperature was raised to 250°F at a rate of approximately 8°F per minute. The pressure and temperature were maintained for one hour. The assembly was then cooled under pressure. These conditions produced a glue line thickness of 2-3 mils. One-inch-wide specimens were cut from the panels with a bandsaw. The pieces from the ends of each panel were discarded.

Specimen Testing

The testing temperatures were maintained by using liquid carbon dioxide and electric heaters as required with a Standard test cabinet. The load was applied with a 60,000-pound Baldwin testing machine operating at a constant rate of crosshead separation. Failure times were measured with a stop watch.

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2. E. McAbee and D. W. Levi, Prediction of Failure Times of Adhesive Bonds at Constant Stress, PATR 4105, in press.

3. B. W. Cherry and C. M. Holmes, Brit. J. Appl. Phys. (J. Phys. D) 2(2), 821 (1969)

TABLE 1

Failure data for AF126 adhesive with 1/8 inch thick aluminum adherends at constant strain rate

Temperature, °K	Crosshead Separation Rate (C), in./min	S, psi	S/T	t_f, min
193	0.105	6100	31.6	1.608
193	0.8	5330	27.6	0.203
193	2	4540	23.5	0.0805
233	0.105	6310	27.1	1.423
233	0.8	5920	25.4	0.198
233	2	5020	21.5	0.065
273	0.105	6300	23.1	1.508
273	0.8	6060	22.2	0.195
273	2	5050	18.5	0.065
296	0.105	5740	19.4	1.332
296	0.8	5490	18.5	0.190
296	2	4870	16.4	0.060
323	0.105	4860	15.1	1.200
323	0.8	4610	14.3	0.160
323	2	4120	12.8	0.053
343	0.105	3790	11.0	1.140
343	0.8	3920	11.4	0.152
343	2	3540	10.3	0.053

TABLE 2

Failure data for AF126 adhesive with 1/16 inch aluminum adherends at constant strain rate

Temperature, °K	Crosshead Separation Rate (C), in. /min	S, psi	S/T	t_f, min
193	0.105	4760	24.7	1.470
193	0.8	4510	23.4	1.428
193	2	4300	22.3	0.108
233	0.105	5410	23.2	1.735
233	0.8	5280	22.7	0.265
233	2	4800	20.6	0.093
273	0.105	5300	19.4	1.657
273	0.8	5160	18.9	0.250
273	2	4660	17.1	0.097
296	0.105	4880	16.5	2.165
296	0.8	4870	16.5	1.747
296	2	4430	14.9	0.108
323	0.105	4320	13.4	1.538
323	0.8	4280	13.3	0.222
323	2	3810	11.8	0.092
343	0.105	2790	8.1	1.893
343	0.8	2520	7.3	0.793
343	2	2750	8.0	0.085

TABLE 3

Deformation from C and t_f values

Temperature, °K	C	$\varepsilon = C\,t_f$ 1/8 inch Al	1/16 inch Al
193	0.105	0.17	0.15
193	0.8	0.16	----
193	2	0.17	0.22
233	0.105	0.15	0.18
233	0.8	0.16	0.21
233	2	0.13	0.19
273	0.105	0.16	0.17
273	0.8	0.20	0.20
273	2	0.13	0.19
296	0.105	0.14	0.23
296	0.8	0.15	----
296	2	0.12	0.22
323	0.105	0.13	0.16
323	0.8	0.13	0.18
323	2	0.11	0.18
343	0.105	0.12	0.20
343	0.8	0.12	----
343	2	0.11	0.17

TABLE 4

Deformation from log C versus log $(1/t_f)$ plots

Temperature, °K	log ε 1/8 inch Aluminum	log ε 1/16 inch Aluminum
193	-0.77	-0.74
233	-0.86	-0.71
273	-0.86	-0.70
296	-0.88	-0.65
323	-0.95	-0.73
343	-0.96	-0.70

TABLE 5

β Values from slopes of the various plots

Temperature, °K	β, Å^3 1/8 inch Aluminum	β, Å^3 1/16 inch Aluminum
	From S Versus Log C Plots	
193	1470	5150
233	2350	4920
273	2880	5610
296	4350	9250
323	5520	8600
343	7790	10400
	From S/T Versus Log C Plots	
193	1480	5150
233	2350	5150
273	3020	5660
296	4230	8650
323	5770	8650
343	9070	11300
	From S Versus Log $(1/t_f)$ Plots	
193	1510	4370
233	2350	4690
273	3010	5270
296	4260	7660
323	5860	7910
343	8900	10400
	From S/T Versus Log $(1/t_f)$ Plots	
193	1480	4120
233	2350	4780
273	2890	5040
296	4230	7570
323	5290	6760
343	9070	11300

TABLE 6

Variation of average β values with temperature

Temperature, °K	β, $A^{\circ 3}$ 1/8 inch Aluminum	1/16 inch Aluminum
193	1500	4700
233	2350	4900
273	2950	5400
296	4300	8300
323	5600	8000
343	8700	11000

TABLE 7

β Values derived from constant stress data

	β, $Å^3$		
Temperature, °K	20% RH	50% RH	95% RH
296	----	----	3800
322	8100	9700	5500
333	9100	10000	7000
344	5200	7800	5900

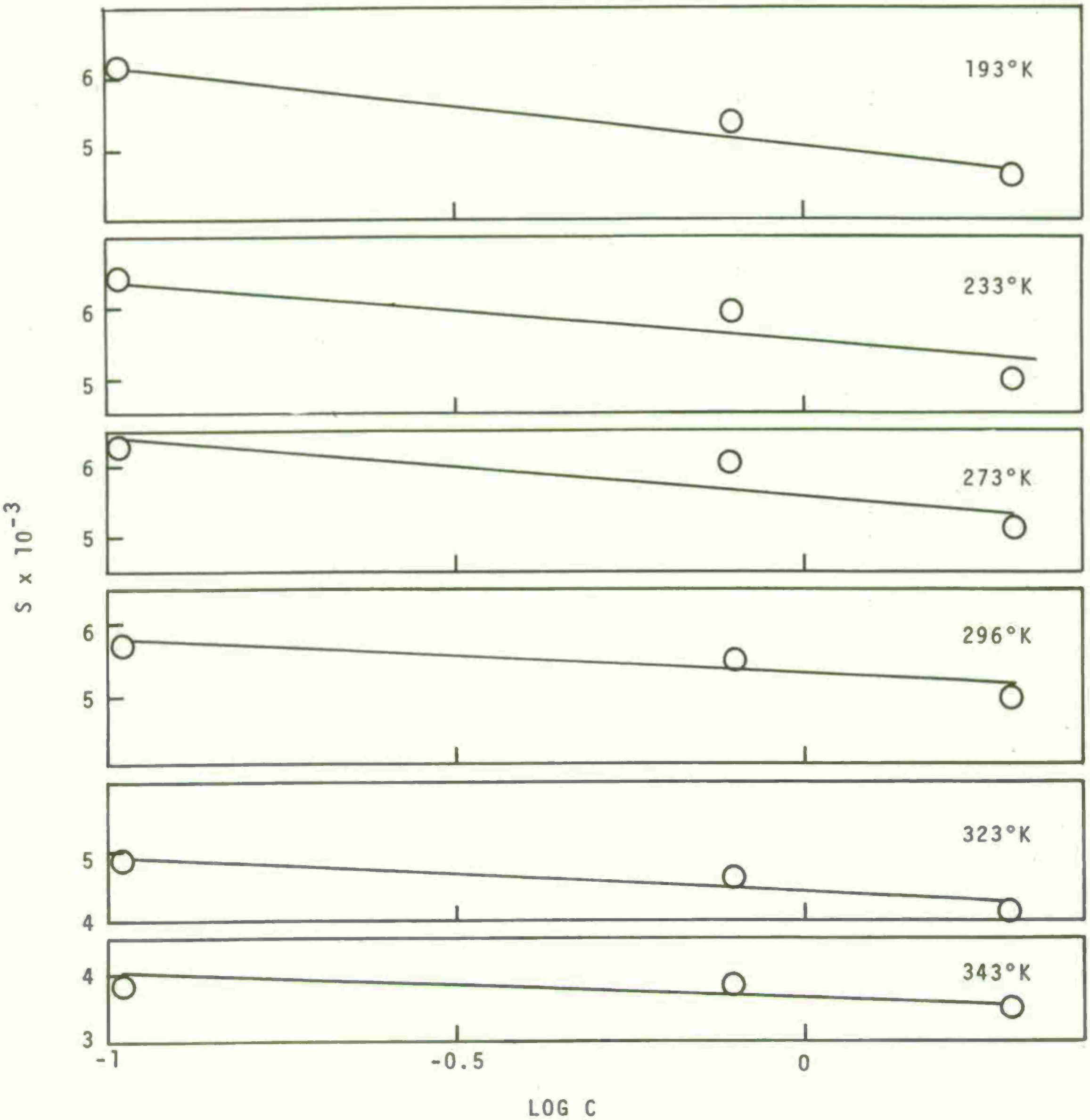

Fig 1 S versus log C for AF126 adhesive with 1/8" aluminum adherends

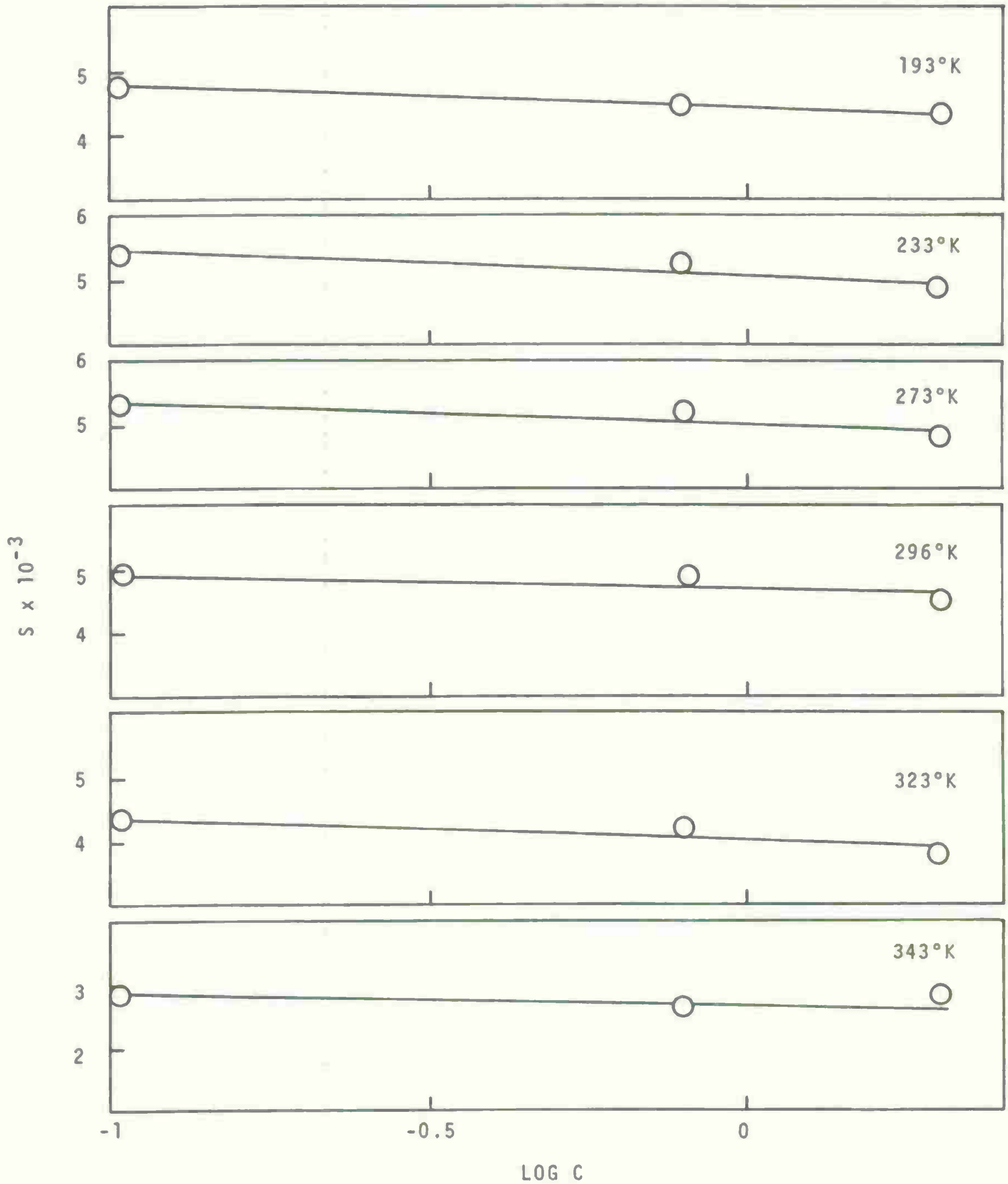

Fig 2 S versus log C for AF126 adhesive with 1/16" aluminum adherends

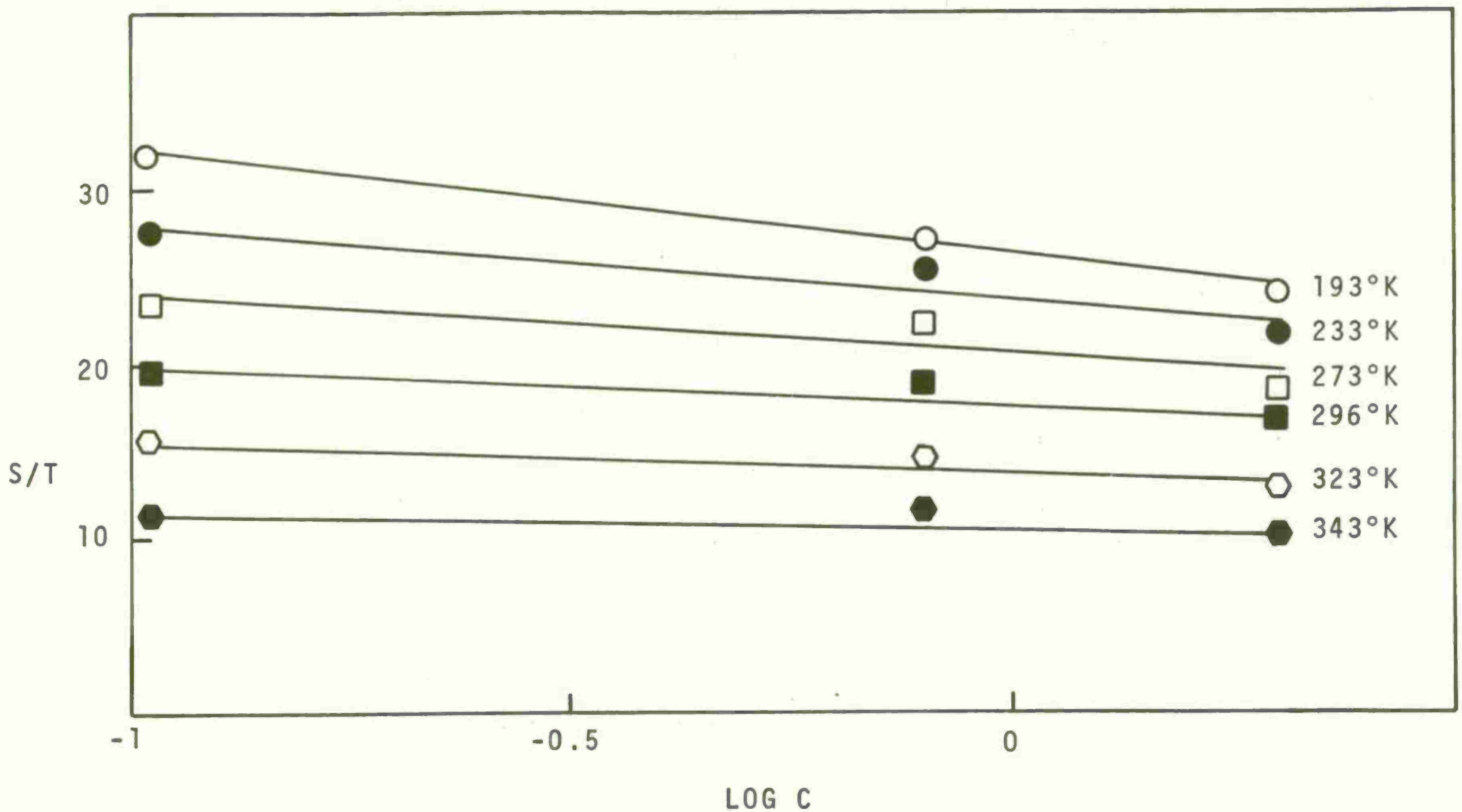

Fig 3 S/T versus log C for AF126 adhesive with 1/8" aluminum adherends

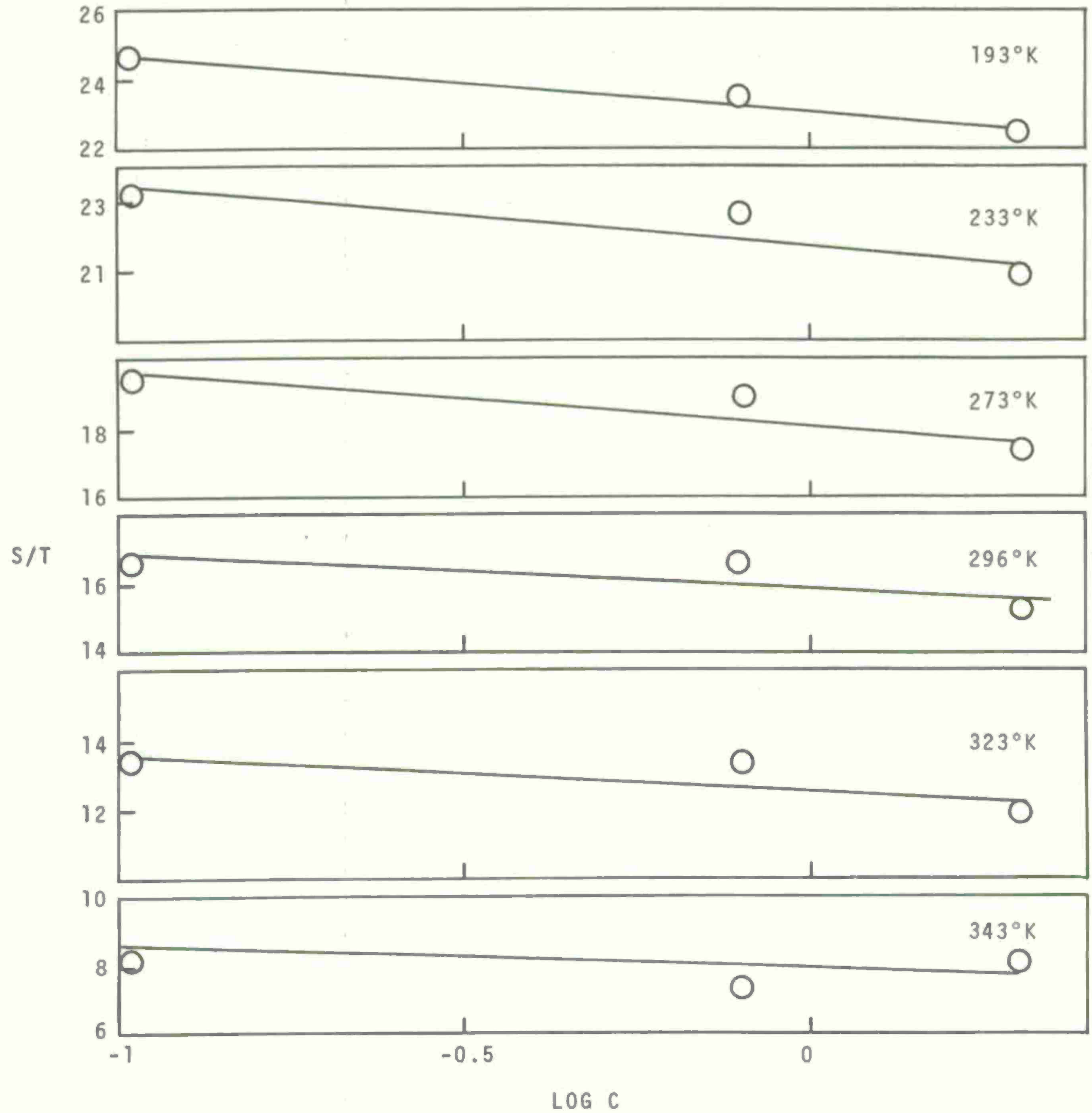

Fig 4 S/T versus log C for AF126 adhesive with 1/16" aluminum adherends

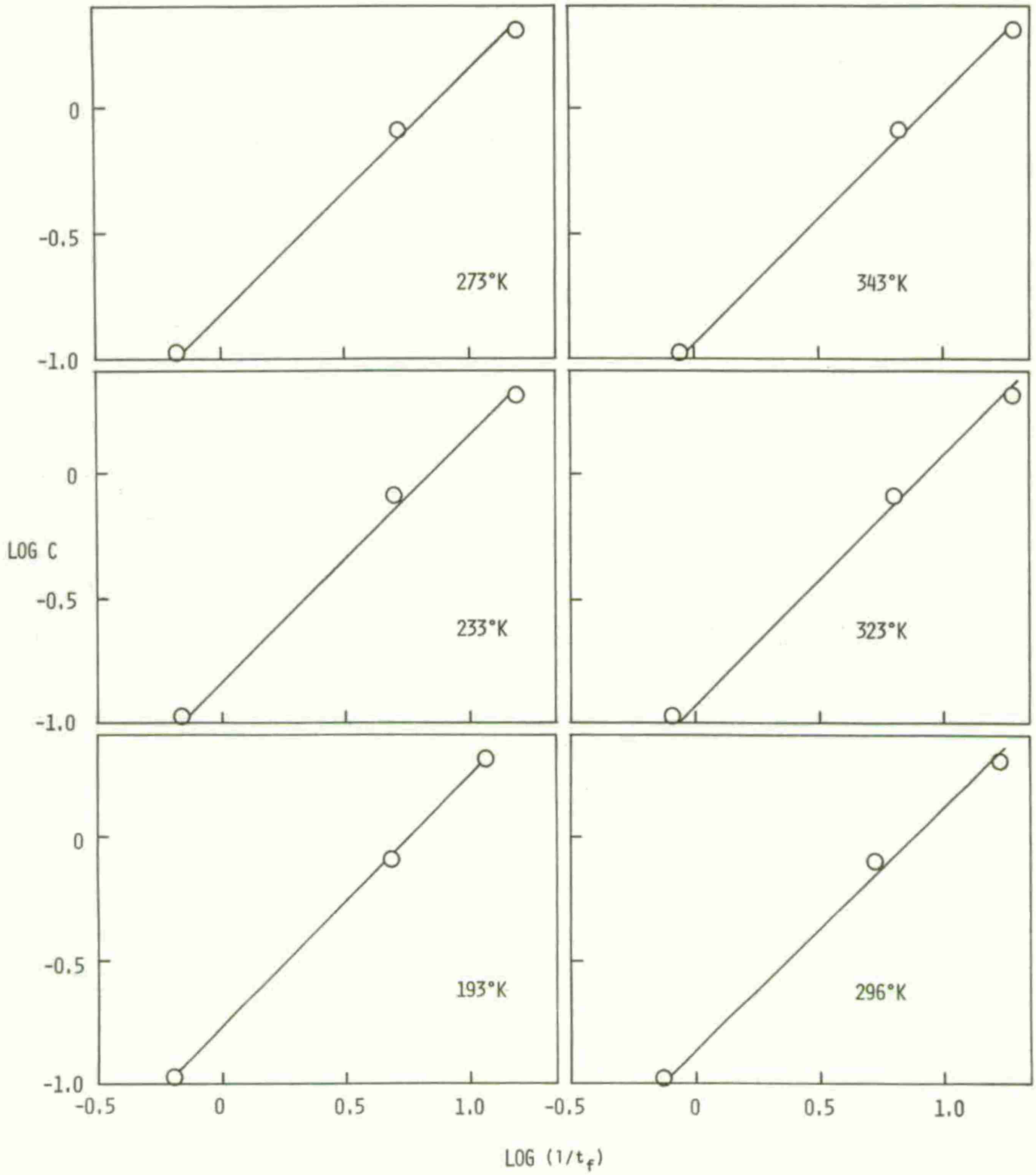

Fig 5 Log C versus log (1/t_f) for AF126 adhesives with 1/8" aluminum adherends

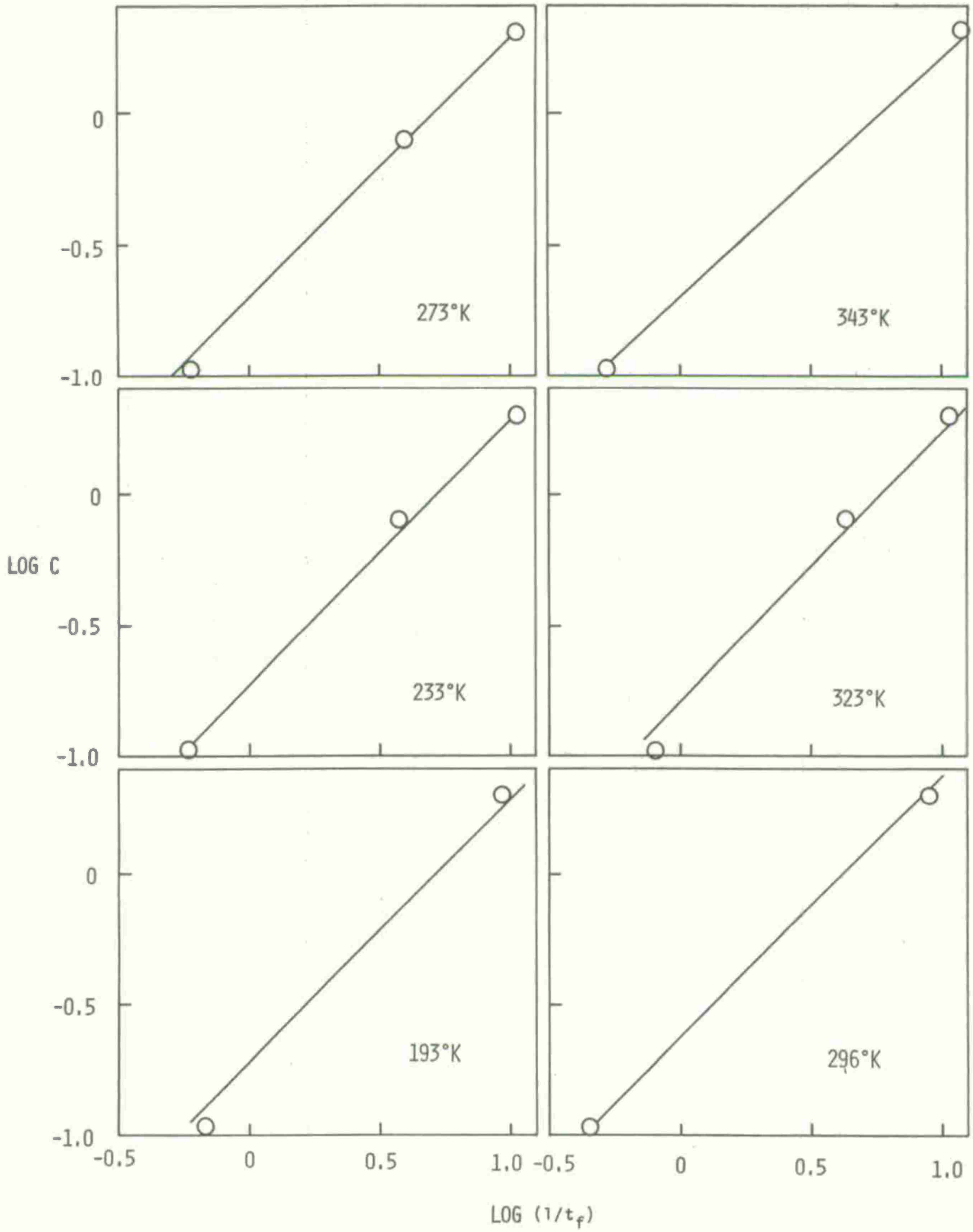

Fig 6 Log C versus log (1/t_f) for AF126 adhesive with 1/16" aluminum adherends

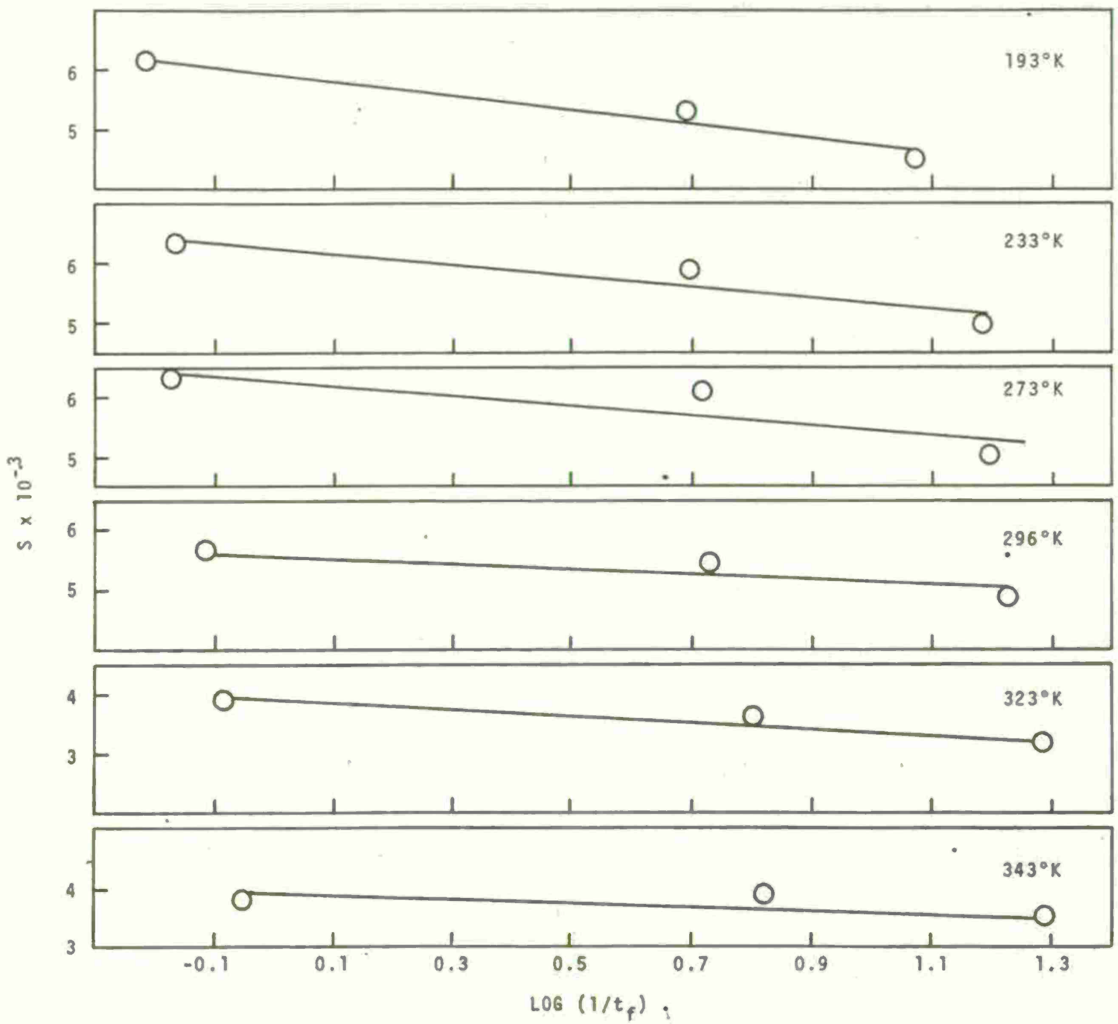

Fig 7 S versus log $(1/t_f)$ for AF126 adhesive with 1/8" aluminum adherends

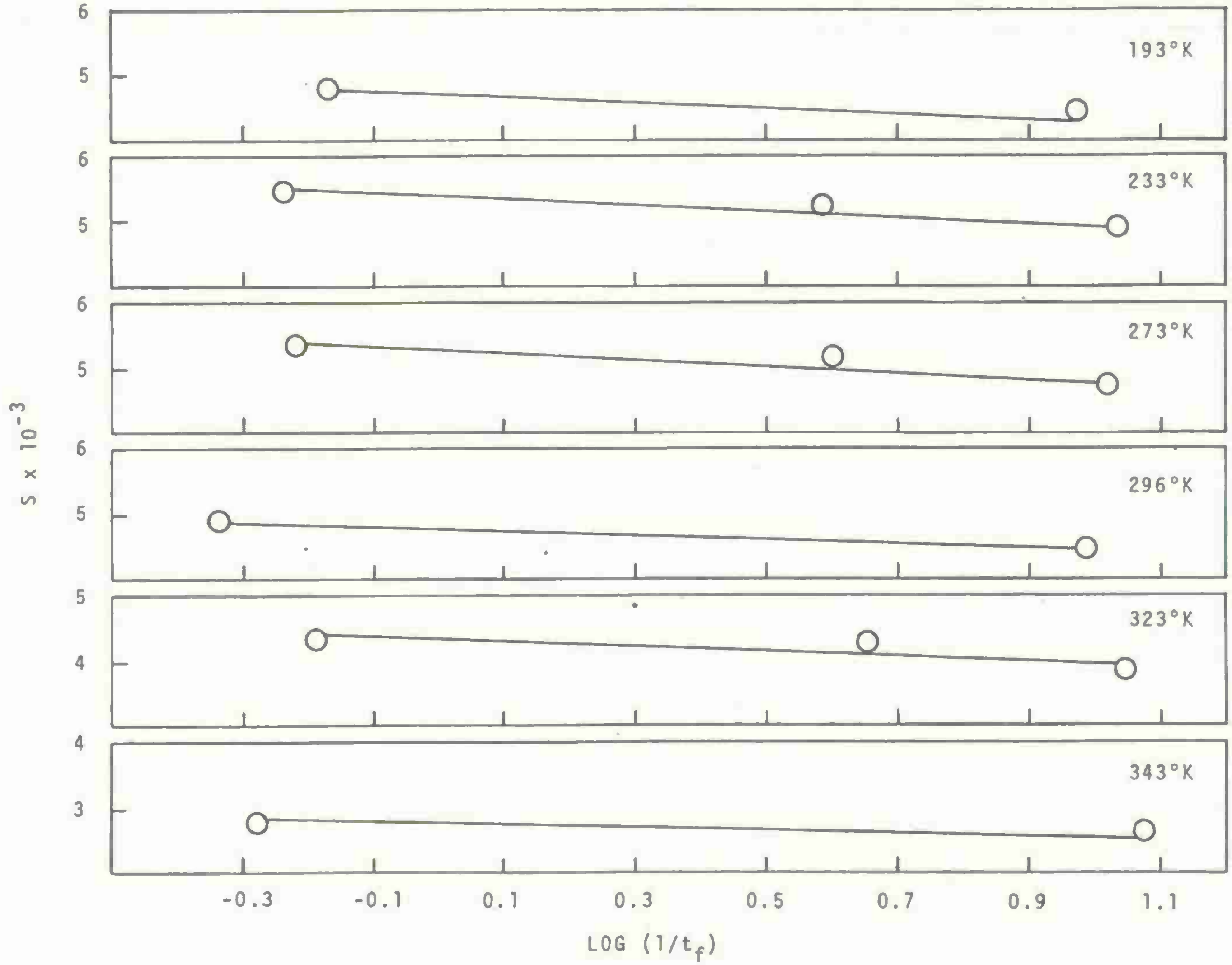

Fig 8 S versus log ($1/t_f$) for AF126 adhesive with 1/16" aluminum adherends

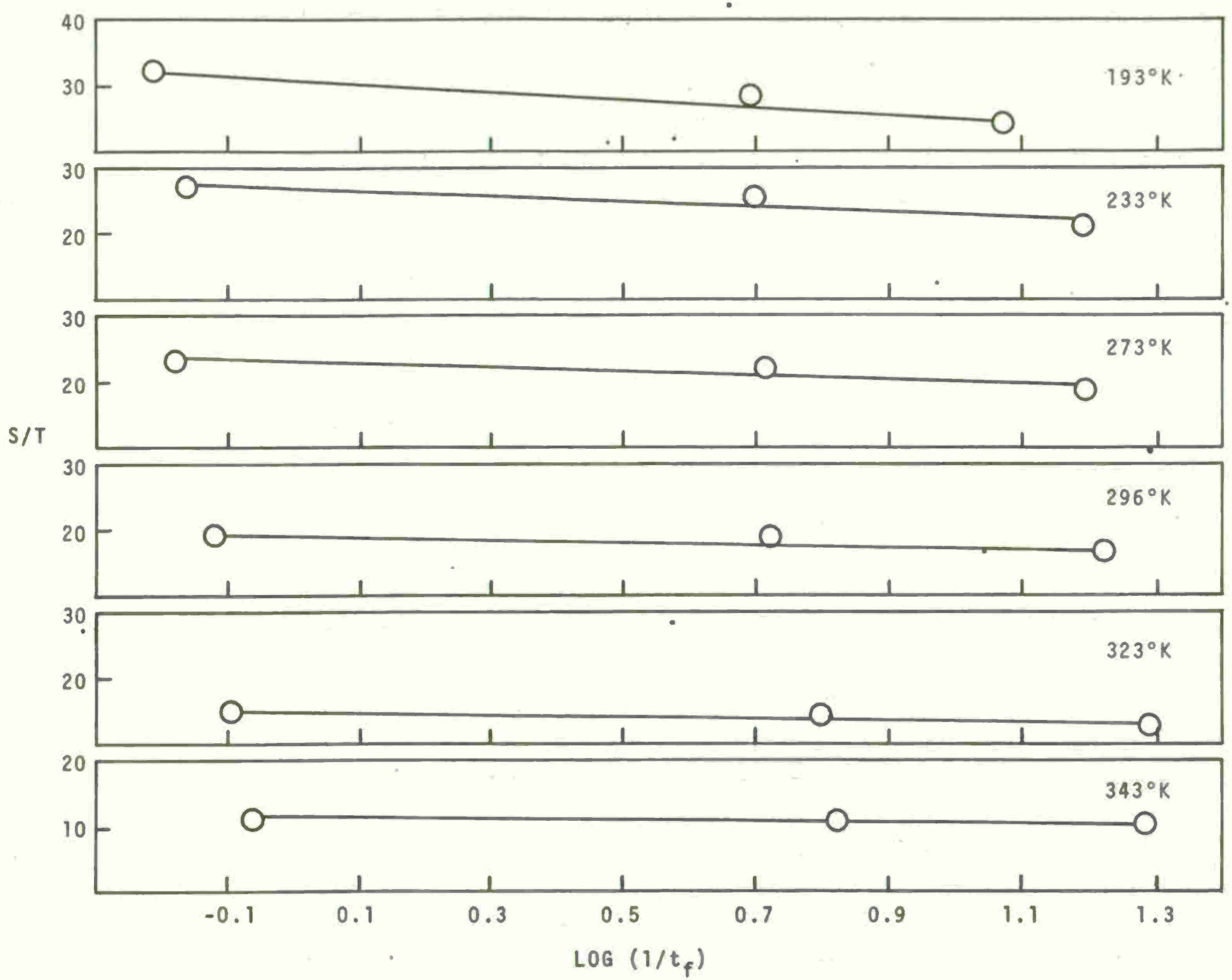

Fig 9 S/T versus log $(1/t_f)$ for AF126 adhesive with 1/8" aluminum adherends

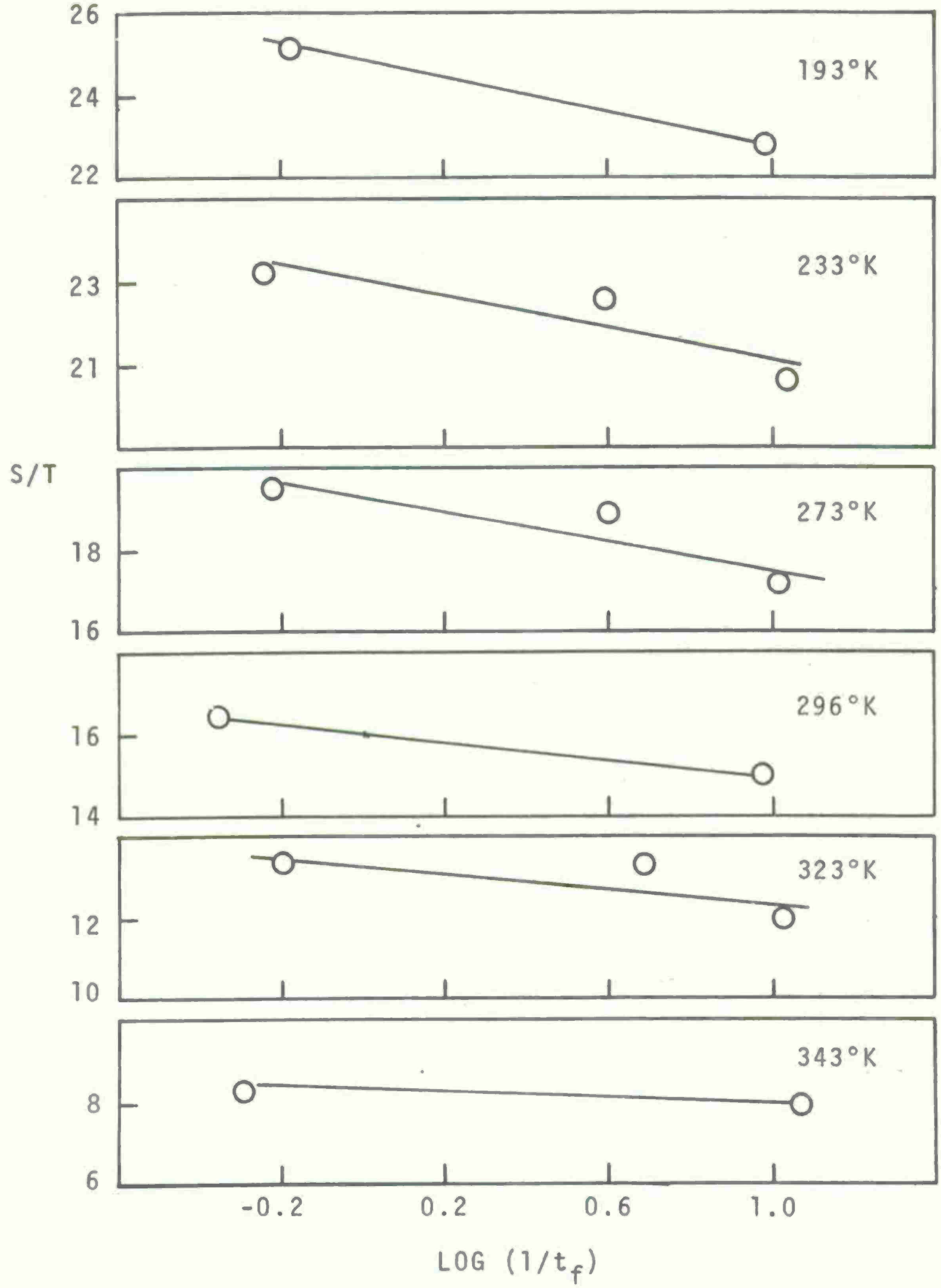

Fig 10 S/T versu log ($1/t_f$) for AF126 adhesive with 1/16" aluminum adherends

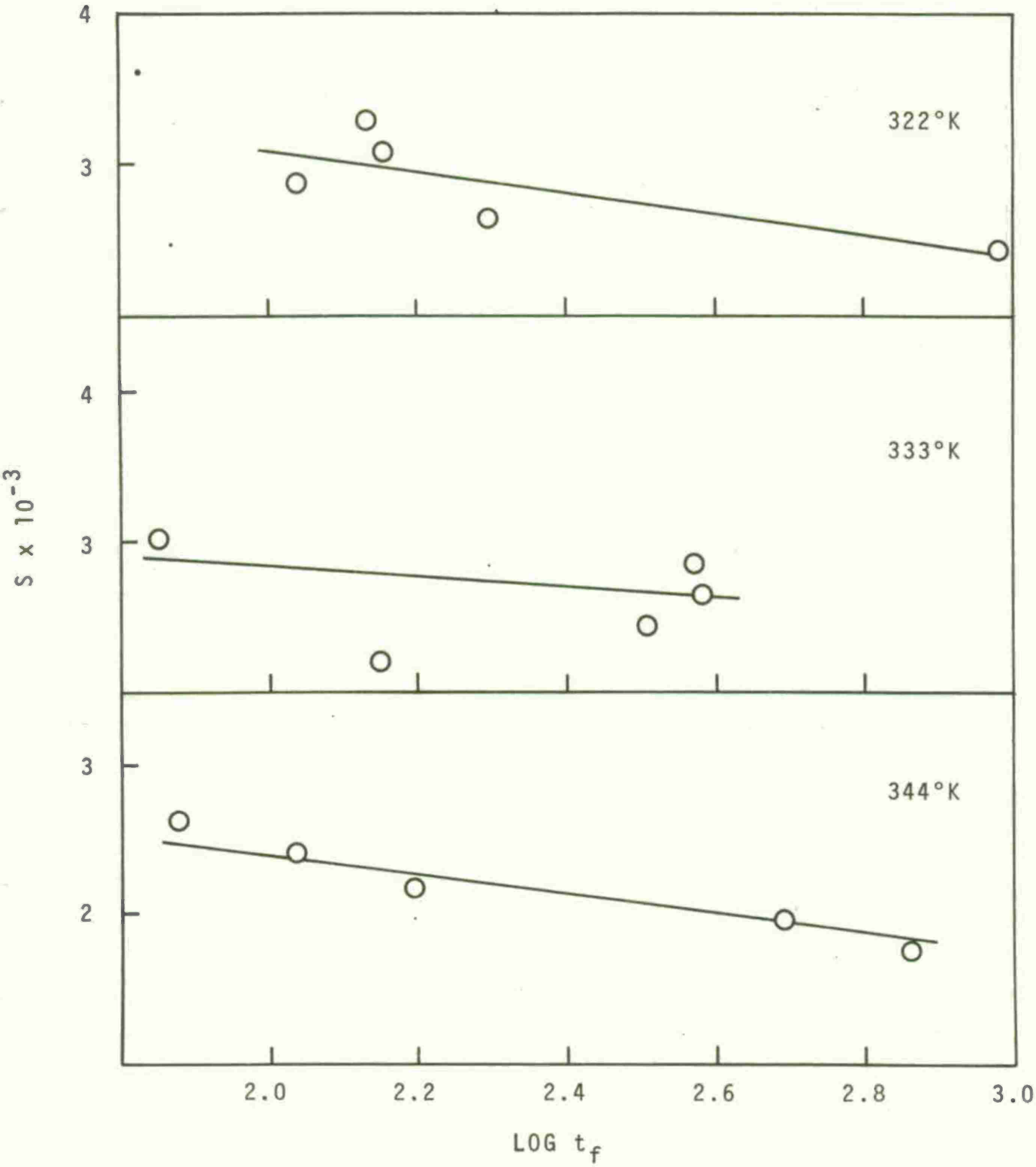

Fig 11 S versus log t_f for AF126 adhesive with 1/16" aluminum adherends 20% Relative humidity

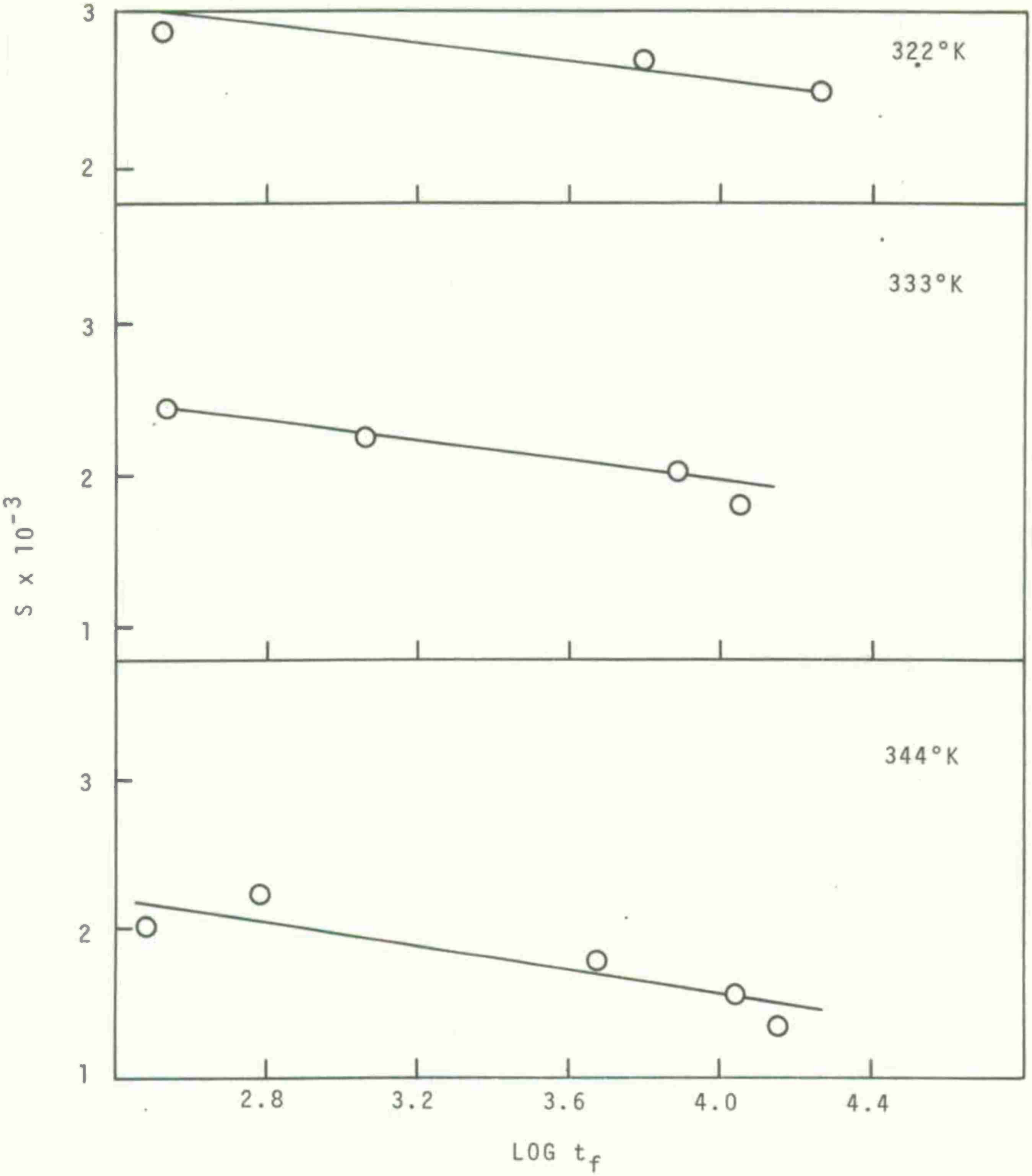

Fig 12 S versus log t_f for AF126 adhesive with 1/16" aluminum adherends 50% Relative humidity

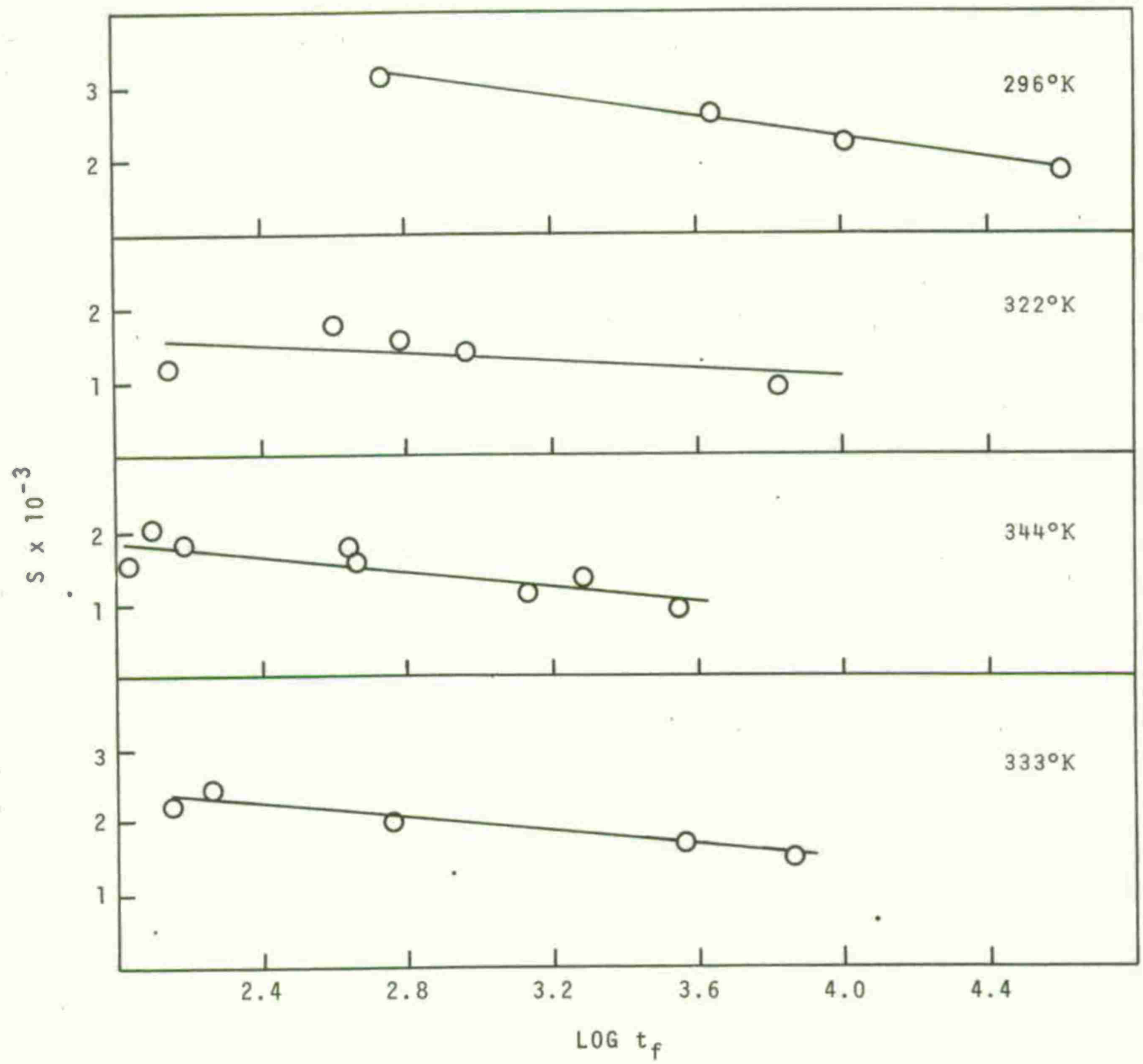

Fig 13 S versus log t_f for AF126 adhesive with 1/16" aluminum adherends 90-95% Relative humidity

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13. ABSTRACT

Shear strengths of adhesive AF126 bonds to two thicknesses of aluminum were measured at constant rate of crosshead separation. Primarily cohesive failure was observed in all cases. By using relations proposed by Cherry and Holmes, the shear strength could be quantitatively related to temperature and strain rate or failure time. Of specific engineering interest would be the possibility of determining the reliability of a bonded joint for specific periods of time as a function of continuously applied stress at a given temperature.

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Adhesive bonds, failure of						
Constant strain rates						
AF126 adhesive						
Aluminum (2024-T3) adherends, 1/8 inch						
Aluminum (2024-T3) adherends, 1/16 inch						
Lap shear specimens						
Temperature effects (193°, 233°, 273°, 296°, 323°, 343°K)						
RH effects (20%, 50%, 90-95%)						
Shear strength of structural adhesives						
Deformation of adherends						
Preparation of adherends						
Preparation of specimens						
Test procedures						